SYNTHESIS AND MECHANICAL BEHAVIOUR OF EPOXY- FLY ASH COMPOSITE

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIRMENT FOR THE DEGREE OF

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By

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National Institute of Technology
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CERTIFICATE

This is to certify that the thesis entitled "**SYNTHESIS AND MECHANICAL BEHAVIOUR OF EPOXY- FLY ASH COMPOSITE**" Submitted by **MR. NITYANANDA KALIA** in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance. To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

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ABSTRACT

The present work describes the development of epoxy based composites with the incorporation of fly ash, an industrial waste, as reinforcement. In coal based thermal power plants fly ash is produced during power generation. Five different volume fractions (viz., 0, 5, 10, 15 and 20% fly ash) were used in the present study to synthesis the epoxy-fly ash composites. Effect of fly ash content on the mechanical properties of these composites under different mechanical test conditions was studied in a comprehensive way. The results suggested that the tensile and compressive strength of these composites increases with increasing fly ash content while the flexural strength and the impact strength decreases with increase in fly ash content. Detailed microscopic observations were carried out for all fractured surfaces.

KEY WORDS: Fly ash, Epoxy resin, Polymer matrix composite, Mechanical behavior

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CHAPTER 1
INTRODUCTION

1.1 Aim of the work: To synthesis and study the mechanical behavior of epoxy – fly ash composite

1.2 Objective of the project: This work involves fabrication of a polymer (Epoxy resin – AW106) composite reinforced with Fly ash particles and its subsequent response to different mechanical behavior. A quick and brief knowledge of the following helps one to get acquainted with the current work and its objectives.

1.3 COMPOSITES

A composite material can be characterized as a mixture of two or more materials that results in preferable properties than those of the individual components. As opposed to metallic combinations, every material holds its different synthetic, physical, and mechanical properties. The two constituents are reinforcement and a matrix. The principle points of interest of composite materials are their high strength and stiffness, combined with low density, when compared with bulk materials**.** Strength and stiffness provided by the reinforcing phase.

The reinforcement is harder, stronger and stiffer than the matrix in most of the cases. The reinforcement is usually a fiber or a particulate. Particulate composites have dimensions that are approximately equal in all directions. They may be spherical, platelets, or any other regular or irregular geometry. Particulate composites are much weaker and less stiff than continuous fiber composites, but they are usually inexpensive. Particulate strengthened composites ordinarily contain less reinforcement (up to 40 to 50 volume percent) because of handling challenges and brittleness.

The fly ash-epoxy composite is a *two-phase* composite where epoxy is the *matrix* and fly ash particles constitute the *dispersed phase.* The matrix is usually referred to the phase that is continuous and surrounds the dispersed phase. A simple classification of the composites based on the type of the reinforcements is given below.

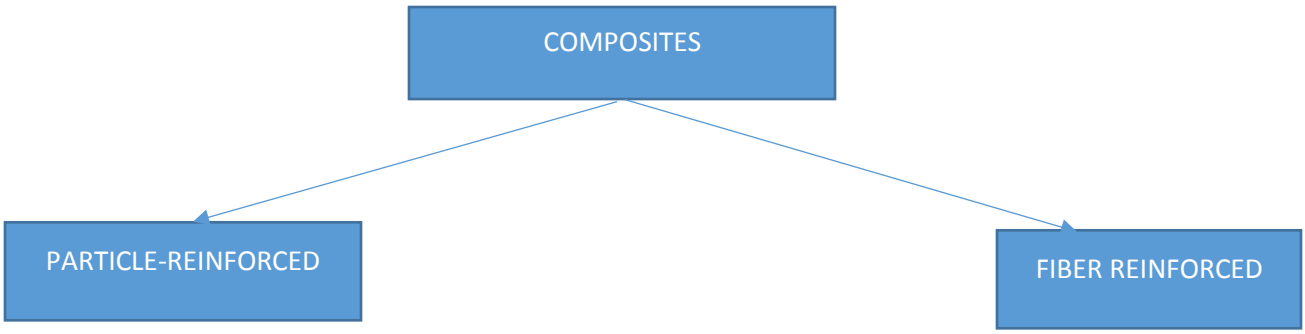


Fig.1.1 Classification of composites

1.4 EPOXY RESIN

Epoxy resin is a class of thermoset materials that extensively used in structural and composite applications because the unique property they deliver is unattainable by other thermoset materials. A wide variety of physical forms, i.e., low viscosity liquid to high melting solid is available in the market. Epoxy system consists of two parts. One part is resin and the other is hardener. When mixed together the resin and hardener activate, causing a chemical reaction which cures the material. Epoxy resin has greater bonding and physical strength than polyester resin. Slow curing property is shown by most of the epoxies. This is widely used as adhesives, coatings, encapsulates, casting materials, potting compounds and binders. Resin and fiber are combined to form complex composite, used in aerospace and recreational industries. Epoxy resins are formulated to generate specific physical and mechanical properties. Emphasis is usually given to tensile strength, modulus and strain, compression strength and modulus, notch sensitivity, impact resistance, heat deflection temperature or glass transition temperature, flammability, durability in service, material availability, ease of processing and price when selecting a thermoset resin. Epoxy resin provides a unique balance of chemical, mechanical properties.

1.5 FLY ASH

Fly ash, otherwise called flue-ash, is one of the residues produced in ignition, and contains the fine particles that ascent with the flue gases. Ash that does not rise is called bottom ash. In a mechanical setting, fly ash typically refers to powder created due to the combustion of coal. Fly ash is for the most part caught by electrostatic precipitators or other molecule filtration component before the flue gases reach the chimney stacks of coal-fired power plants, and together with bottom ash expelled from the base of the furnace is for this situation mutually known as coal ash. However all fly ash incorporates considerable measures of silicon dioxide (SiO_2) (both amorphous and crystalline) and calcium oxide (CaO), both being endemic ingredients in numerous coal-bearing rock strata.

Fly ash is a pozzolanic material. It is a finely-separated alumino-silicate with different amounts of calcium, which when blended with portland concrete and water, will react with the calcium hydroxide discharged by the hydration of portland cement to create different calcium-silicate hydrates (C-S-H) and calcium-aluminate hydrates. Some fly ashes with higher measures of calcium will likewise show cementitious conduct by responding with water to produce hydrates without a source of calcium hydroxide. These pozzolanic reactions are valuable to the solid in that they build the amount of the cementitious binder phase (C-S-H) and, to a lesser degree, calcium-aluminate hydrates, enhancing the long-term quality and diminishing the permeability of the system. Both of these instruments upgrade the durability of the cement.

CHAPTER 2

LITERATURE REVIEW

T. Chaowasakoo et al. **[1]** have studied the effect of conventional thermal and microwave curing methods on mechanical and morphological properties of fly ash/epoxy composites. The conventional thermal curing was carried out at 70^0C for 80 min. while the microwave curing was done at 240 W for 18 min in order to get the optimum cure of the composite. The tensile, flexural moduli of the fly ash/epoxy composite increased with increase in the fly ash content while the mechanical strength (tensile, flexural and impact strength) continuously reduced with increase in fly ash content. Up to 0.5 wt. % coupling agent can be added to the fly ash/epoxy composite for the enhancement of the mechanical properties.

Caifen Wang et al.[**2**] worked on the effect of fly ash cenospheres on the microstructure and properties of silica based composites. They found that the density of the sample decreased on increasing the amount of fly ash cenospheres while the strength increased inversely when the content of the sphere was lower than 50wt%. Fly ash cenospheres acts as a sintering aid to densification of silica and restrain the crystallization of cristoballite as an inhibitor. The growth of the mullite from sphere to matrix forms a powerful framework for the final product.

Pradeep Sambyal et al. [**3**] work was based on advanced anticorrosive properties of polymer/fly ash composite. By the help of epoxy resin, the copolymer composite coatings were developed. FTIR, XRD, TGA and SEM techniques were used to characterize the coatings. A significant improvement in the thermal stability of epoxy coatings with the addition of copolymer composite was shown by TGA analysis. A decrease in current density was clearly shown in the corrosion results and SEM micrographs of the coated surfaces with defects. Low corrosion current for coatings with 2.0 and 3.0 wt.% loading of copolymer composite in 3.5 wt.% NaCl solution indicated by the Tafel parameter. Superior corrosion protection property was shown by these coatings even for prolong exposure to 3.5 wt. % NaCl solution.

D.A. Papargyris et al. **[4]** studied mechanical and physical properties of a carbon fiber epoxy composite manufactured by resin transfer molding using conventional and microwave heating microwave transforming holds extraordinary potential for enhancing current composite assembling strategies, generously decreasing cure process durations, vitality necessities and operational expenses. In this paper, microwave warming was fused into the gum exchange forming system. Through the utilization of microwave warming, a 50% cure process duration decrease was attained to. The mechanical and physical properties of the delivered carbon fiber/epoxy composites were contrasted with those fabricated conventional resin transfer molding**.**

Venceslav Vassilev and *et al.* **[5]** work on Composites Containing Waste Materials and they find that the composite materials grew on the premise of unsaturated polyester tars indicate generally great quality attributes and can discover application in the machine-building industry for the creation of lodgings and different parts, supplanting different materials with comparative parameters, yet of higher expense. They additionally find that the materials taking into account epoxy resin are better electro protectors and could be effectively utilized for electrical protection mixes with application in electrical developments, radio-gadgets hardware and different sorts of electrical gear and machines

CHAPTER 3

EXPERIMENTAL PROCEDURES

3.1 Measurement of Pycnometry Density of fly ash:

It was done by pycnometric method. In this method fly ash was poured into a 10 ml bottle. Fly ash was taken one third volume of this bottle. Then the bottle was filled up to half of its volume and then the bottle was kept in vacuum for removal of the bubble. This procedure was followed for 2 hours. After that weights were taken

Let the weight of empty bottle= W_1=20.7971gm

Let the weight of sample+ bottle = W_2 =24.5697gm

Let the weight of bottle + sample + water = W_3 = 47.3378gm

Let the weight of bottle + water = W_4 = 45.6610gm

Now the actual weight of the sample = (W_2-W_1) gm = 3.7726gm

Weight of Liquid (in this case water) = (W_4-W_1) gm =24.8639gm

Weight of water other than sample volume = (W_3-W_2) gm = 22.7681gm

Now the specific gravity of sample is given by

$$[(W_2-W_1)/ (W_4-W_1)- (W_3-W_2)]$$

$$=1.8$$

So the density of the sample = specific gravity of the sample × Density of water at room temperature

$$= 1.8 \times 0.9999720 \text{ g/cm}^3$$

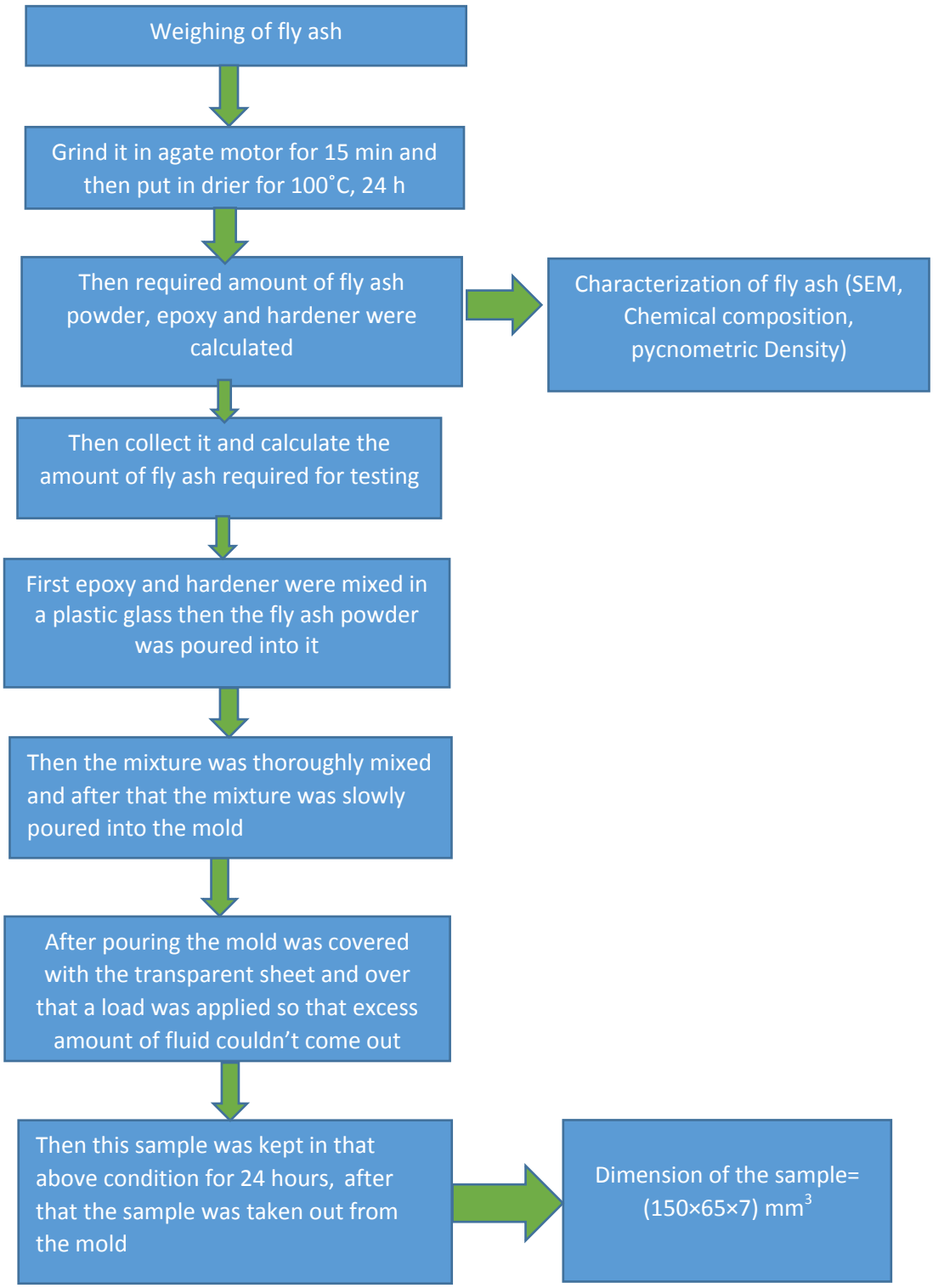
$$= 1.799\text{g/cm}^3 \approx 1.80\text{g/cc}$$

The density of the waste fly ash was found to be =1.8 g/cm^3.

3.2 Detailed procedure for making epoxy-fly ash composite

Fly ash particles were collected from the workshop, Ceramic department, was grounded for 15min with the help of agate motor to form a fine powder. Then this fly ash was kept in a drier for 100˚C, 24 hours to remove the moisture present in it. After 24 hr. the fly ash was taken out from the drier. Then the amount of fly ash, epoxy resin (AW106) and hardener (HY 953) was calculated for five different composites (0, 5, 10, 15 and 20 vol% fly ash).

FLOW CHART EXPERIMENTAL PROCEDURE



Required amount of epoxy, hardener and fly ash were mixed properly in a plastic glass with the help of a spoon. It was mixed till a little amount of heat was generated that indicated the reaction between fly ash and the polymer matrix started. The mixture was mixed for 10 min to form a homogenized mixture. Then the mixture was casted using a mold made up of wooden bit, the size of the mold is 150 X 65 X 7 mm^3 **(Fig. 3.1)**. The diamond spray was sprayed on the transparent sheet (before pouring the viscous mixture) so that the composite material wouldn't stick to the transparent sheet. While pouring the mixture inside the mold, care should be maintained so that the fly ash–epoxy mixture wouldn't come out of the mold. Then again a transparent sheet with diamond spray spread on it was covered on the surface of the mold to prevent the contaminated particles from outside. A roller was rolled on the surface of the sheet to make the surface flat. The castings are left to cure at room temperature for about 24 h after which the wooden bits are broken and sample was released.

3.2.1 Calculation of required fly ash powder, epoxy resin and hardener:

3.2.1.1 Tensile and flexural testing sample preparation:

Fly ash density (obtained from pycnometry) = 1.8 gm/cc

Epoxy resin (**AW 106**) density = 1.17gm/cc

Hardener (**HY953**) density = 0.95 gm/cc

Now the volume of the mold= 150mm×65mm×7mm = 68250 mm^3

The ratio of epoxy to hardener added = 10:8

So the percentage of hardener = (8/18) × 100 = 44.44%

The percentage of epoxy = (10/18) × 100 = 55.55%

Now the density of matrix was given by,

ρ_{matrix} = (44.44/100) × 0.95 + (55.55/100) × 1.17

= 1.072 gm/cc

a) for 0 vol% fly ash,

Fly ash required = 0 gm

Mass of matrix = 1.072 × 68250 × 10^{-3} gm

= 73.16 gm

Now mass of epoxy required = (10/18)×73.16 gm = 40.64 gm

Mass of hardener required = (8/18) × 73.16 gm = 32.51 gm

b) for 5 vol%fly ash,

Fly ash required = $1.8 \times 0.05 \times 68250 \times 10^{-3}$ gm = 6.1425 gm

Mass of matrix = $0.95 \times 1.072 \times 68250 \times 10^{-3}$ gm

= 69.5058 gm

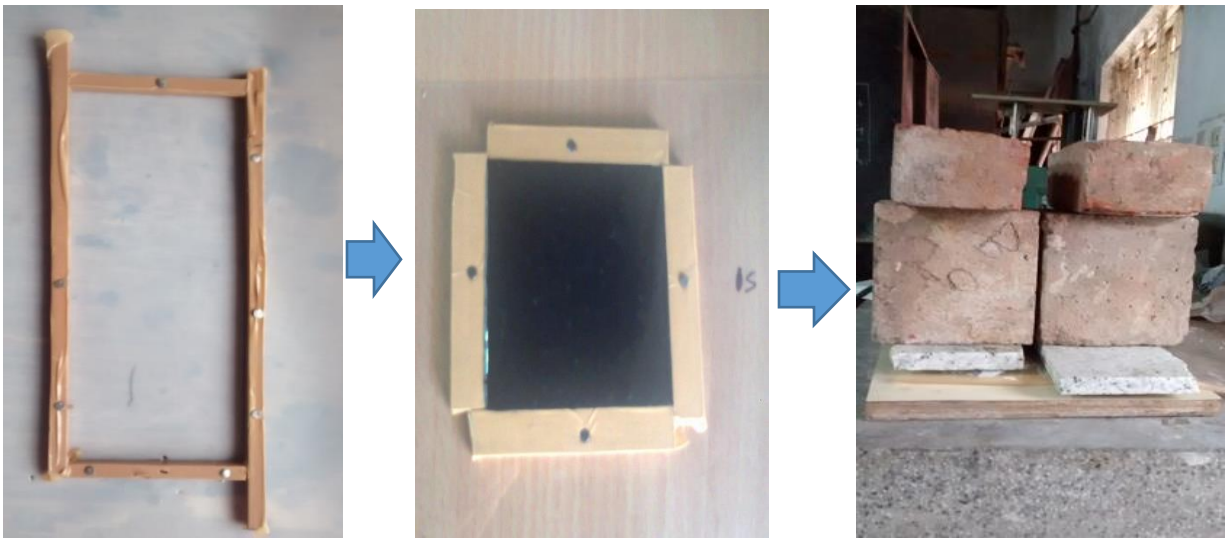
Now mass of epoxy required = (10/18) × 69.5058 gm = 38.62 gm

Mass of hardener required = (8/18) × 69.5058 gm = 30.88 gm

Other three vol% of fly ash (i.e. 10, 15 and 20 vol %) was also calculated in a similar manner

Vol% of fly ash	Wt. of fly ash (in gm)	Wt. of epoxy (in gm)	Wt. of hardener (in gm)
0	0	4.0298	32.24
5	6.14	38.62	30.88
10	12.28	36.59	29.26
15	18.43	34.55	27.64
20	24.57	32.51	26.01

Table 3.1 Required amounts of fly ash, epoxy and hardener for tensile and flexural test



(a) **(b)** **(c)**

Fig. 3.1 Basic 3 steps process for casting the epoxy fly ash composite

The above cast slab dimension mentioned was about tensile and flexural test. The slab of a rectangular bar shape of dimension 150x65x7 mm^3 was prepared. After that we cut half of the slab, so that one half was used for flexural test and other half was used for tensile test. As a result two rectangular bar shaped slabs of equal dimensions 75x65x7 mm^3 were obtained. For each half we made four rectangular shape bars of equal width 13 mm. For tensile test we made those four bars into a dog bone shape piece of gauge length 40 mm. Its width and thickness was measured. For flexural test we took the four rectangular bars and its width, thickness were measured. Similar process was carried out for other composite specimens with varying amount of fly ash.

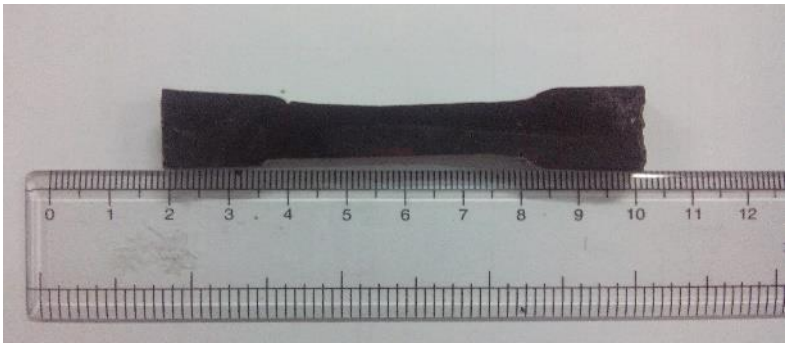


Fig 3.2 Dog bone sample for tensile test

Fig 3.3 Sample for flexural test

Fig 3.4 Flexural test apparatus

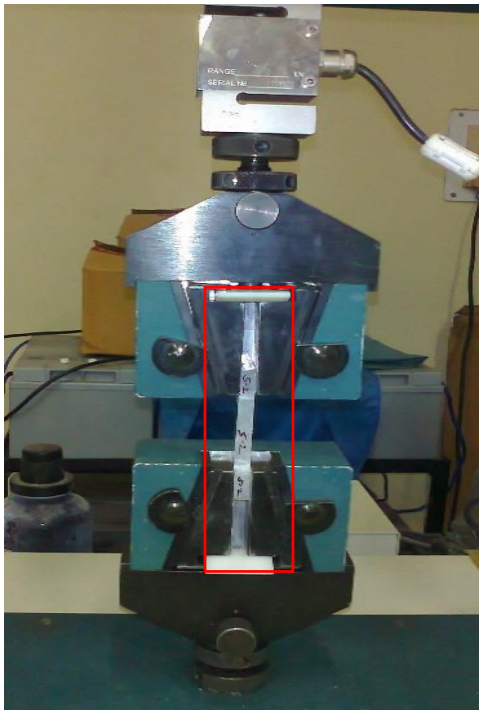


Fig-3.5 Tensile test apparatus

3.2.1.2 Compression testing sample preparation

For compression test a completely different process was used. Here we took a pipe of inner dia. approx. 17mm. Then we cut the pipe into equal heights of apprx.21mm. Then we covered the bottom portion of the pipe into a cello tape so that when we poured the fly ash- polymer matrix mixture from the top it wouldn't come out at the bottom. Before casting the inner wall of the pipe was lined with vaseline jelly so that the composite wouldn't stick into the pipe walls. The casting was left to cure for 24 h. After that the sample was taken out. Filing needs to be done on both the surfaces of the sample. For each vol% of fly ash we made 3 samples for compression test. Sample dia. and height is 17 mm. i.e. l/d=1.

Calculation for compression mold was given below.

Diameter of the mold = 17 mm; Height taken = 22mm,

Volume of mold = $\pi \times \frac{d\text{^}2}{4} \times h$ = 4993.56mm^3

where d=diameter of mold and h= height of mold

a) for 0 vol% fly ash,

Fly ash required = 0 gm

Weight of matrix = 1×1.072×4993.56×10^{-3} gm = 5.353gm

Mass of epoxy = (10/18) ×5.353gm = 2.973gm

Mass of hardener= (8/18) × 5.353gm = 2.379gm

b) for 5 vol% fly ash,

Fly ash required = 0.05x1.8×4993.56×10^{-3}gm =0.449 gm

Weight of matrix = 0.95×1.072×4993.56×10^{-3} gm= 5.085gm

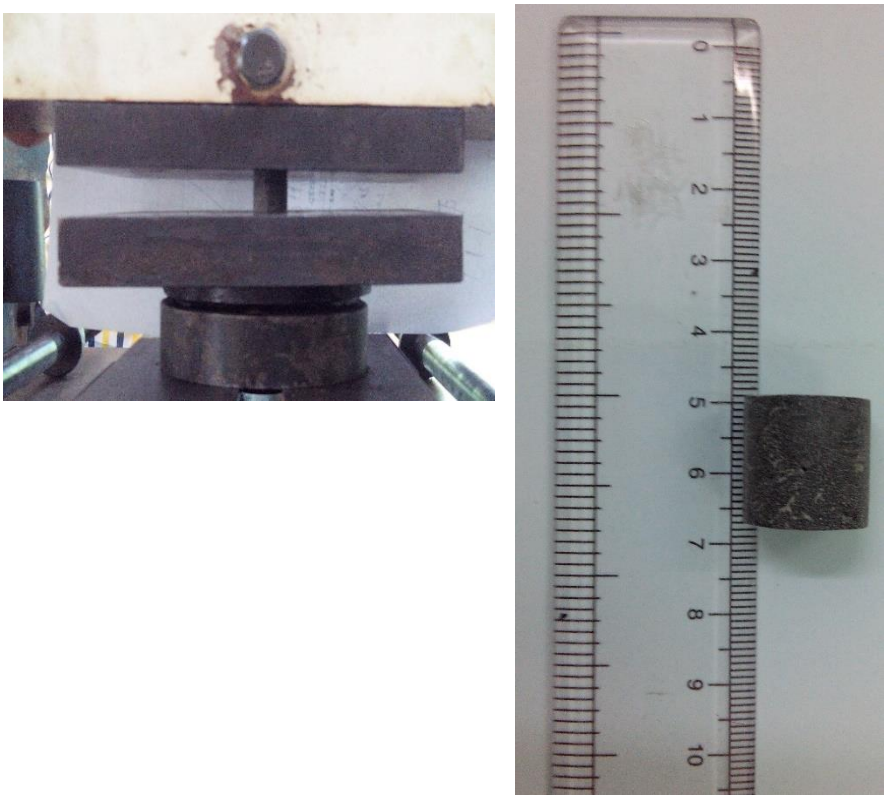
Mass of epoxy would be = (10/18) × 5.085gm = 2.825gm

Mass of hardener would be= (8/18) × 5.085gm = 2.26gm

Similarly other 3 vol% of fly ash (10,15 and 20 vol%) was calculated.

Vol % of fly ash	Wt. of fly ash (in gm.)	Wt. of epoxy (in gm.)	Wt. of hardener (in gm.)
0	0	2.973	2.379
5	0.449	2.825	2.26
10	0.898	2.676	2.142
15	1.348	2.527	2.022
20	1.798	2.379	1.903

Table 3.2 Required amounts of fly ash, epoxy and hardener for compression test



(a) ***(b)***

Fig 3.6 (a) shows the compression test set-up (b) shows the sample for the compression test

3.2.1.3 Impact testing sample preparation:

The dimension of the mold would be taken as 65mm×65mm×5mm.

Calculations for impact mold were given below:

Length of the mold = 65mm;

Breadth of the mold = 65mm;

Height of the mold = 5mm;

Now the volume of the mold = 65mm×65mm×5mm = 21125mm^3

Now a) for 0 vol% fly ash,

Fly ash required = 0 gm

Weight of matrix = 1×1.072×21125×10^{-3} gm = 22.452gm

Mass of epoxy would be = (10/18) × 22.452gm = 12.473gm

Mass of hardener would be= (8/18) × 5.066gm = 9.978gm

Now b) for 5 vol% fly ash,

Fly ash required = 0.05×1.8×21125×10^{-3} gm = 1.9gm

Weight of matrix = 0.95×1.072×21125×10^{-3} gm = 21.51gm.

Mass of epoxy would be = (10/18) × 21.51gm = 11.95gm

Mass of hardener would be= (8/18) × 21.51gm = 9.56gm

Similarly other 3 vol% of fly ash (10, 15 and 20 vol %) was calculated.

Vol % of fly ash	**Wt. of fly ash (in gm)**	**Wt. of epoxy (in gm)**	**Wt. of hardener (in gm.)**
0	0	12.47	9.97
5	1.9	11.95	9.56
10	4.56	13.58	10.86
15	6.84	12.83	10.26
20	9.12	12.08	9.66

Table 3.3 Required amounts of fly ash, epoxy and hardener for impact test

Fig 3.7 Impact test apparatus

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Characterization of fly ash

4.1.1 SEM

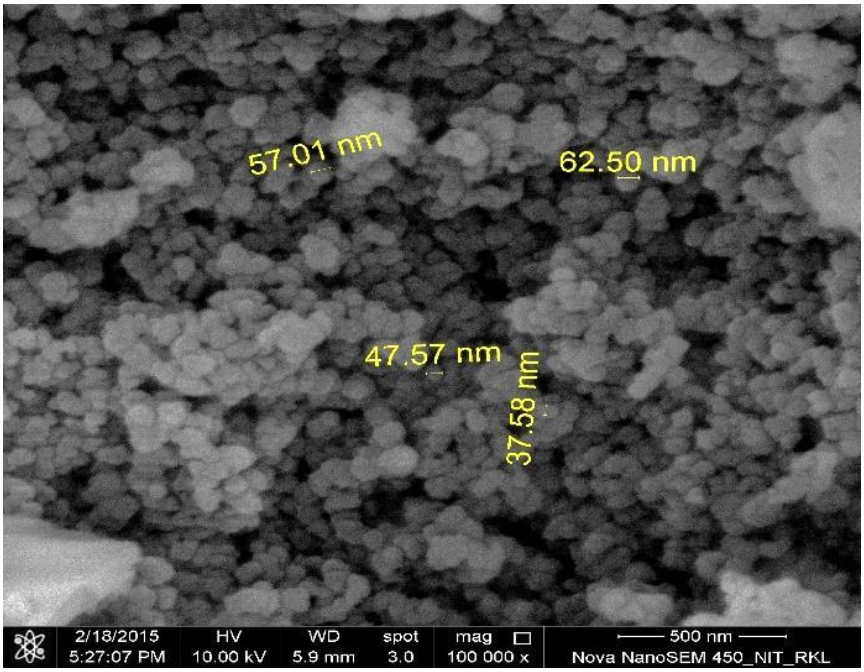


Fig 4.1 SEM of fly ash shows rounded morphology of fly ash particles

4.1.2 Chemical analysis Report

COMPOSITION	PERCENTAGE (wt%)
Fe_2O_3	1.326
CaO	2.32
MgO	0.55
SiO_2	94.01
Al_2O_3	1.77

Table 4.1- Chemical analysis of fly ash

Pycnometric density of fly ash is 1.8 g/cc

4.2. DENSITY MEASUREMENT OF THE COMPOSITE

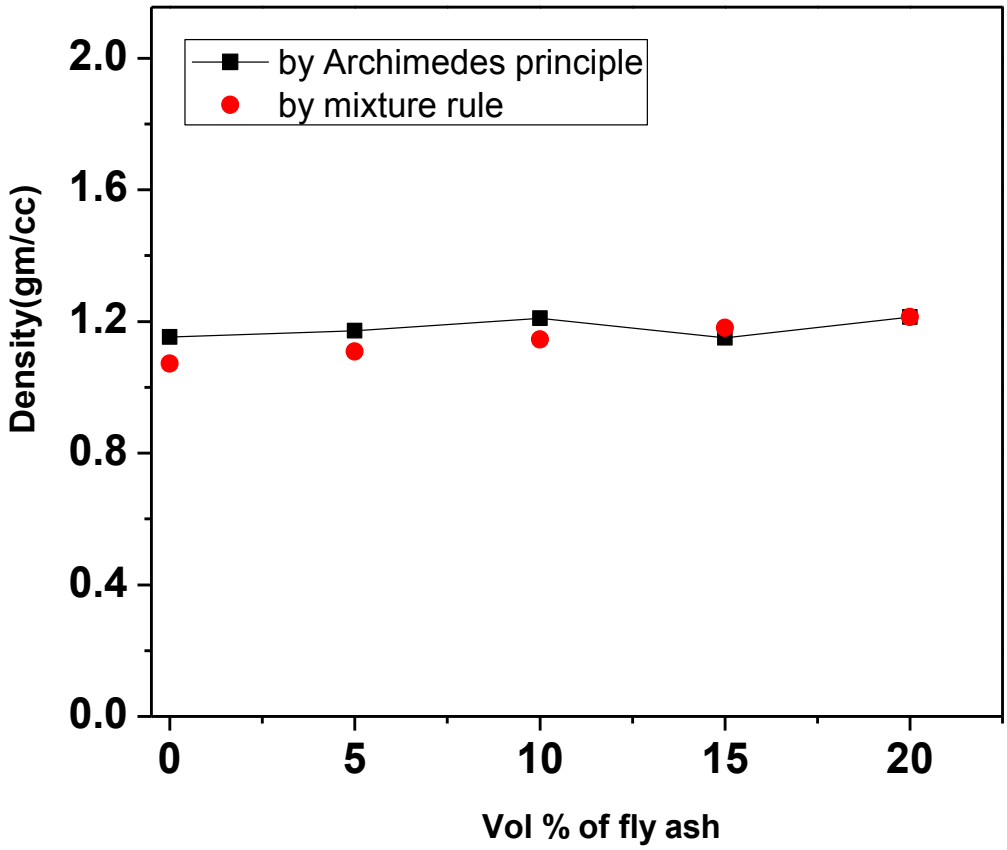
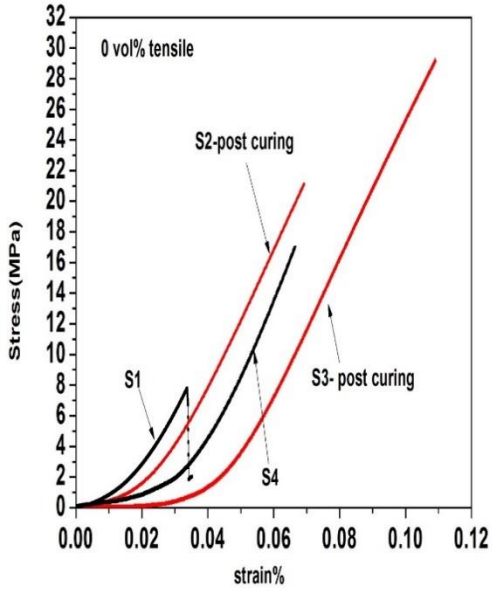


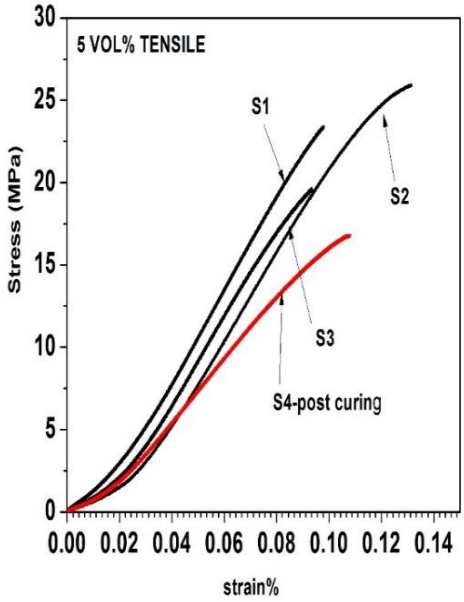
Fig- 4.2: Variation of Archimedes density with fly ash content.

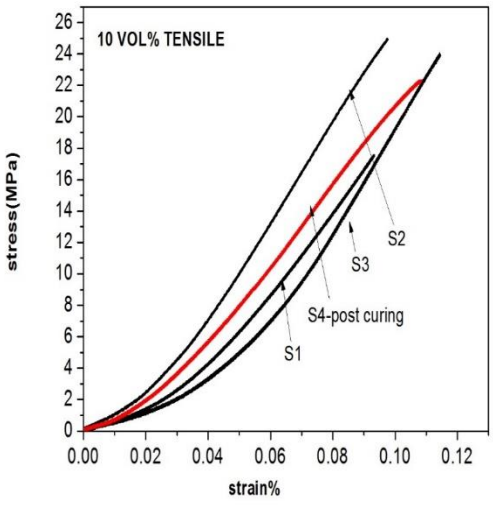
From the fig 4.2 shows that the density of the sample was measured by two different ways, i.e (i) by Archimedes method and (ii) by rule of mixture. In both way densities are increased due to the incorporation of fly ash into the epoxy resin that results in increase in weight of the sample, along with the increment of the addition of fly ash content.

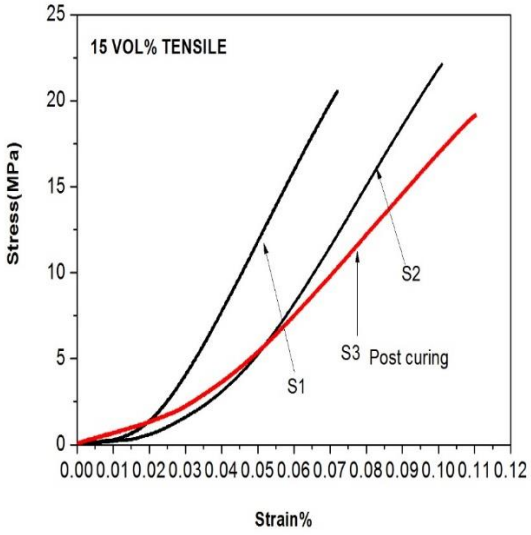
4.3 Mechanical testing

4.3.1Tensile test









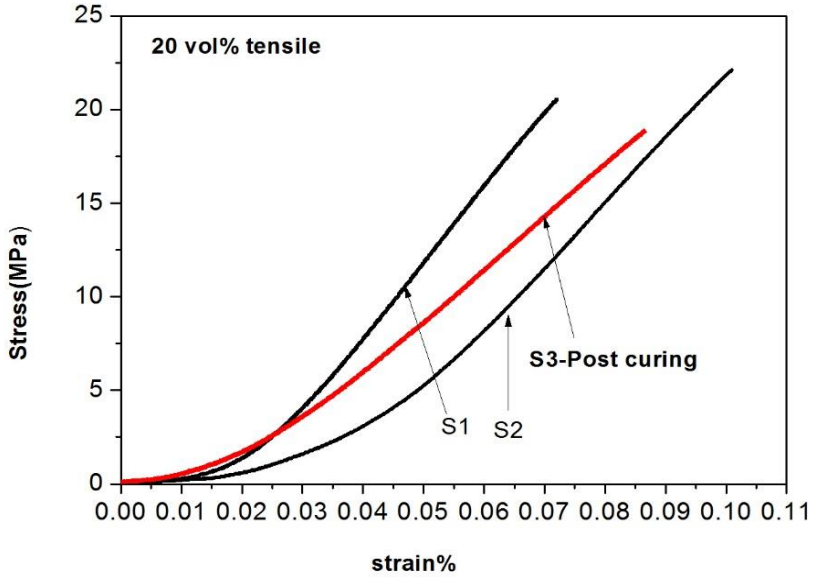


Fig-4.3 Results for tensile testing for different vol% of fly ash epoxy composite (each having 4 samples). Red color depicts samples after post-curing.

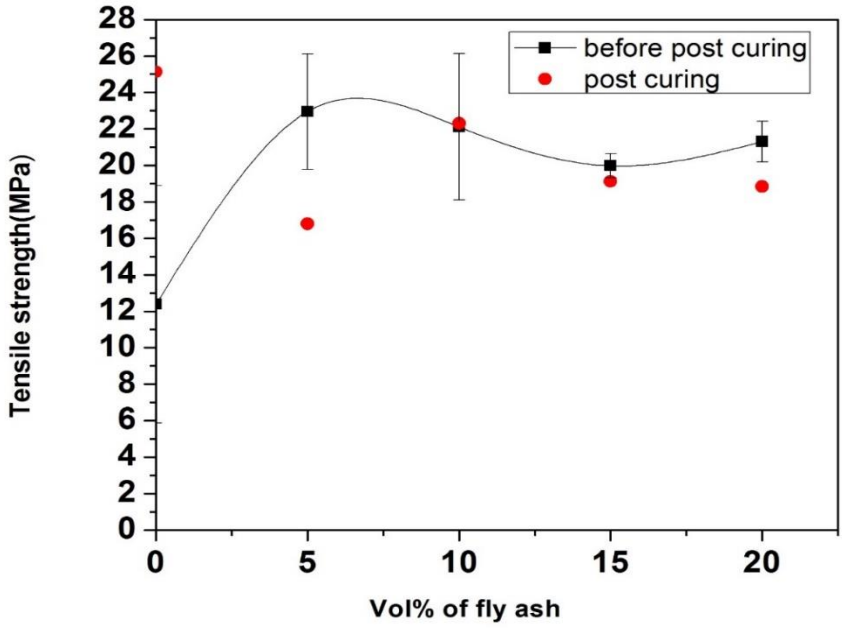
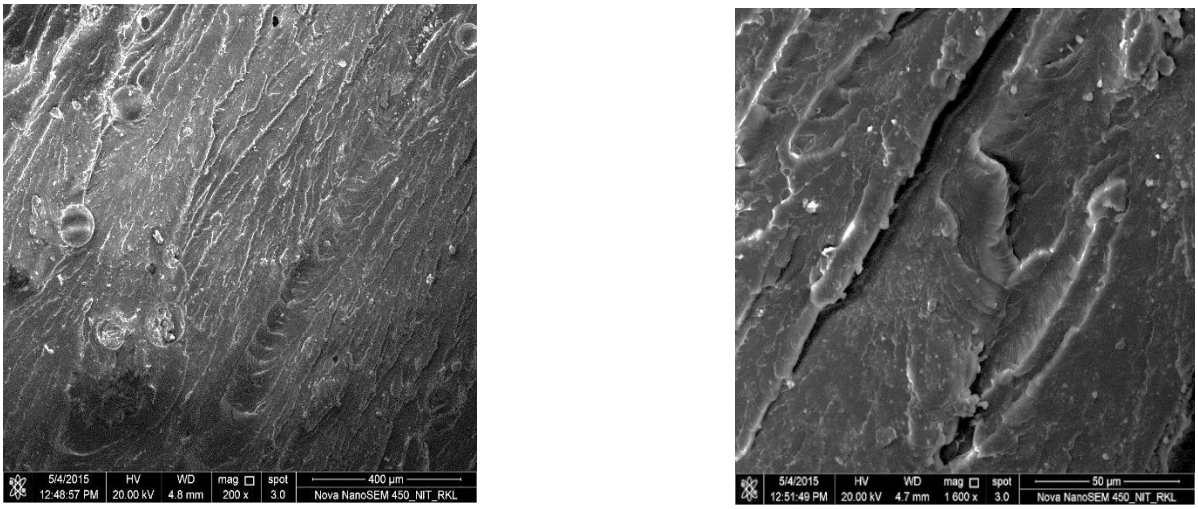


Fig-4.4 Variation of tensile strength with the content of fly ash.

The red mark indicates the post curing effect of the sample i.e. the samples were kept in drier at 100˚C for 15 min. From the above Fig.4.3 and Fig. 4.4 we saw that in 0 vol% sample 1 has very less stress. It is due to the sample was broken from its neck part. From fig. 4.4 it is observed that the tensile strength initially increases then decreases. It is due to the fact of the strong interface bonding between the fly ash and the polymer matrix composite up to 5 vol%, but then the tensile strength decreases probably due to the wetting phenomena. According to wetting phenomena upon addition of high amount of fly ash, the reinforcement (viz., fly ash) is not wet properly by the polymer (epoxy) matrix. As a result poor interface bonding occurred between fly ash and polymer matrix that results decrease in strength.

4.3.1.1 MICROSTRUCTURE AFTER TENSILE TEST-

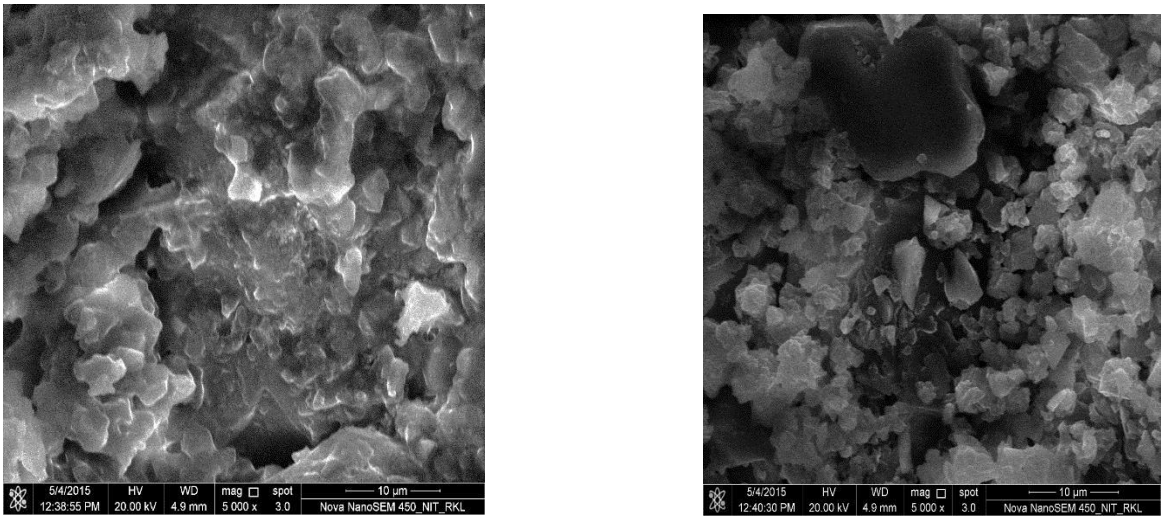
0 VOL%



(a) ***(b)***

Fig 4.5 SEM images of fracture surface of 0 vol% fly ash-epoxy composite after tensile testing

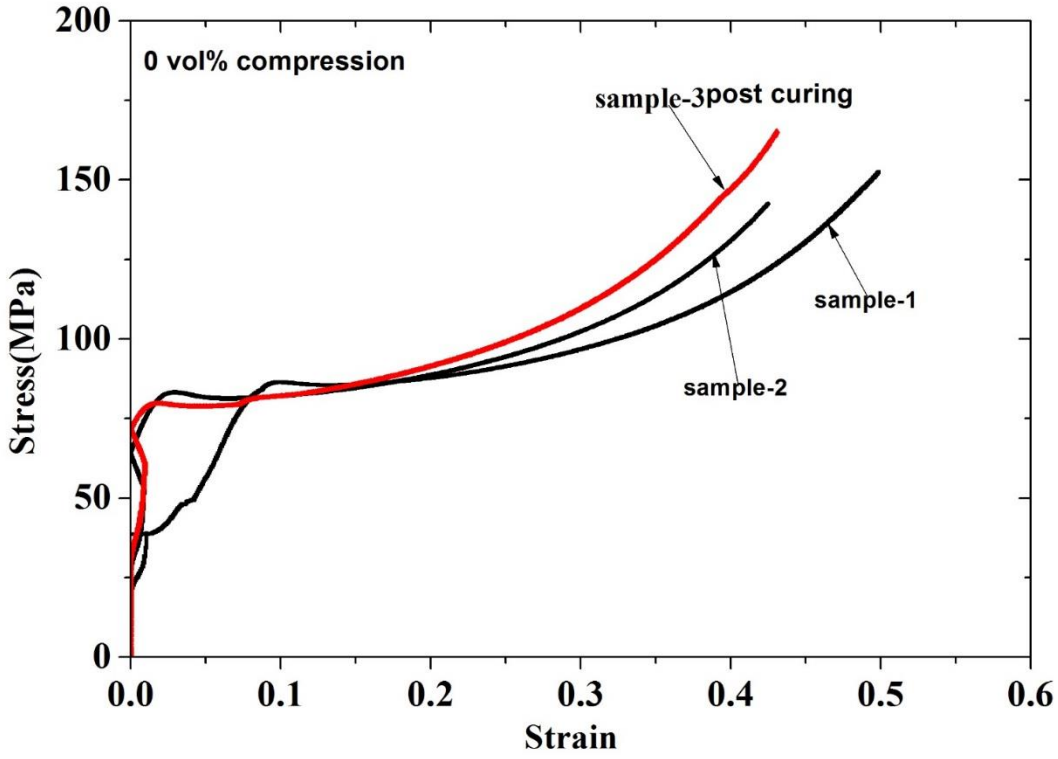
20 VOL%



(a) ***(b)***

Fig 4.6 SEM image of fracture surface of 20 vol% fly ash –epoxy composite after tensile testing

4.3.2 COMPRESSION TEST



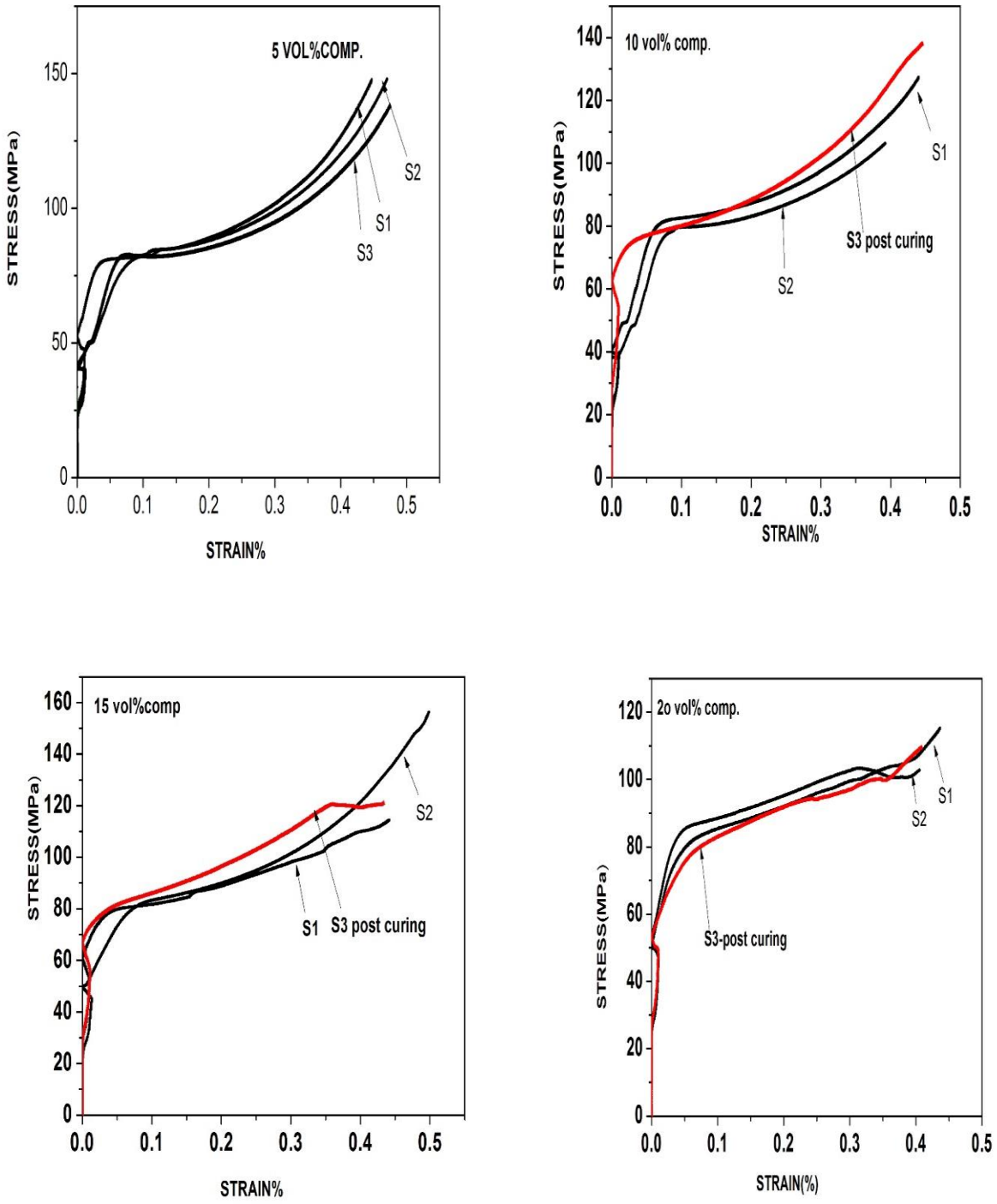


Figure-4.7: The above graphs show the strees vs strain variation for each vol% of fly ash

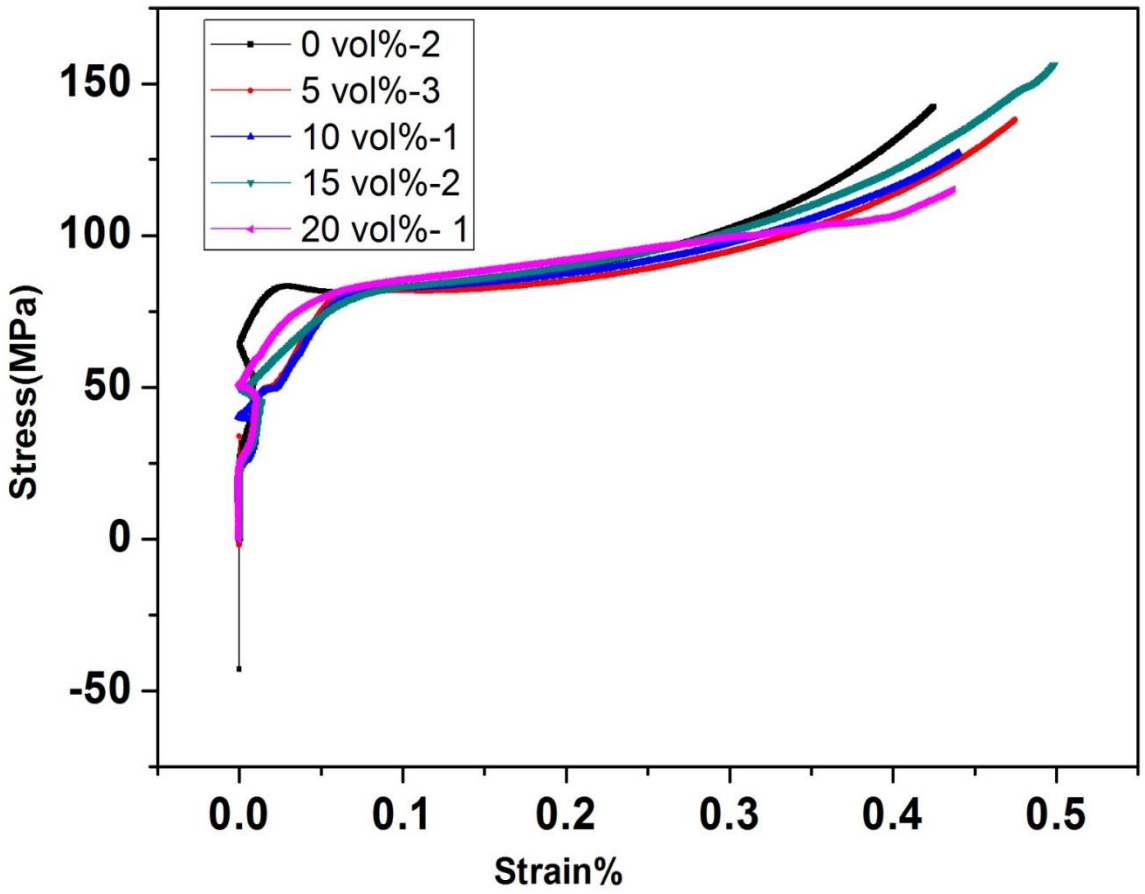


Fig. 4.8: Composite graph for Stress vs strain curves for different vol% of fly ash

From fig 4.8 it is observed that stress increases on increasing fly ash content. It is probably due to the strong interface between fly ash and epoxy matrix composite**.**

4.3.3 Impact test

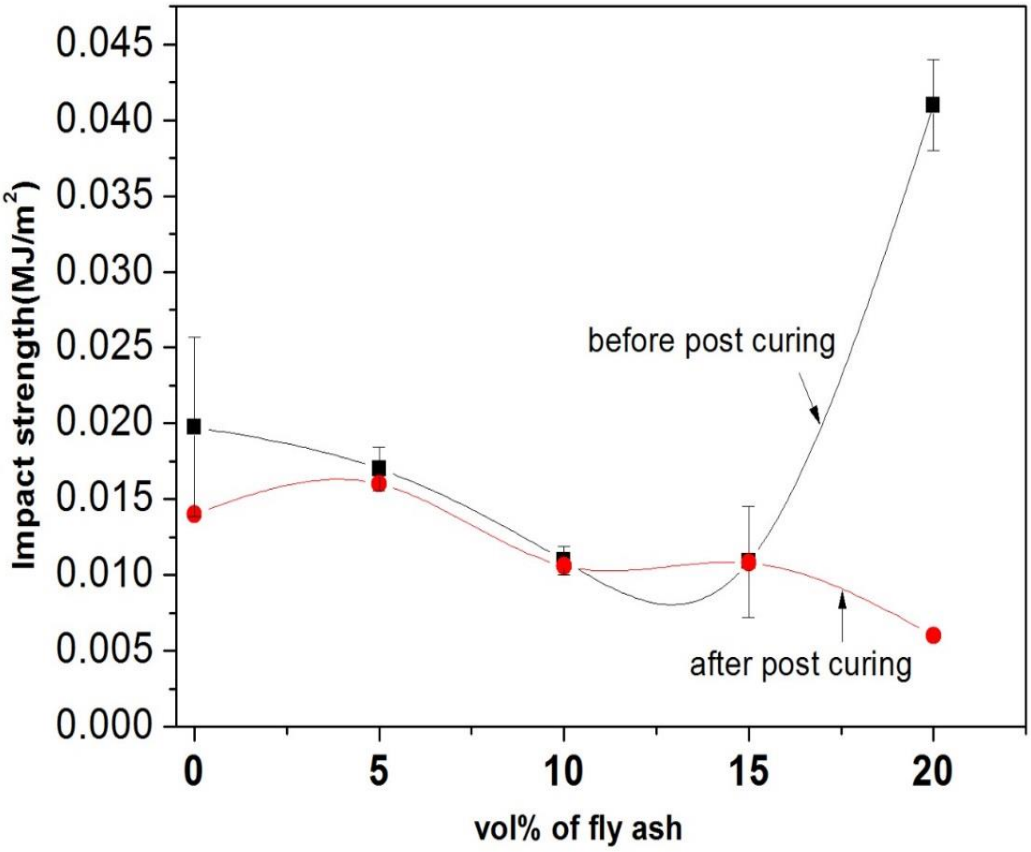
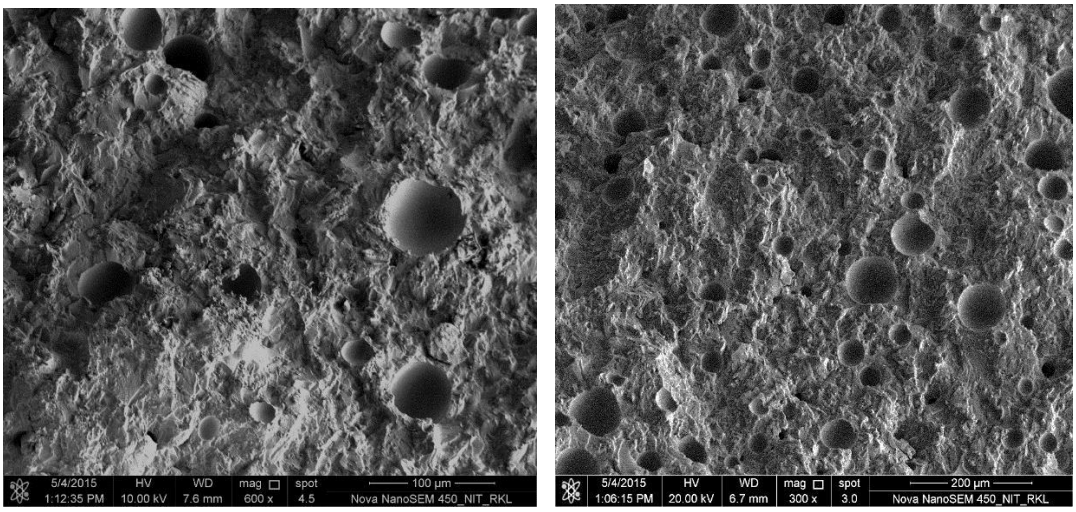


Fig- 4.9: graph between Impact Strength vs Vol% fly ash

The red mark indicates the post curing effect of the sample i.e the samples were kept in drier at 100˚C for 15 min. From the fig.4.9 above we saw that on increasing fly ash amount, the impact strength decreased up to 15 vol% then it increased abruptly at 20 vol%. Similarly in post curing the tensile strength increased slightly up to 5 vol% then it decreased. The reason was conventional cured composite has a large temperature gradient across the specimen thickness. Due to large temperature gradient, some internal stresses developed inside the sample causing lowering of the impact strength.

4.3.3.1MICROSTRUCTURE AFTER IMPACT TEST-



(a) ***(b)***

Fig 4.10 SEM images of fracture surface of 20 vol% fly ash – epoxy composite after impact test

4.3.4 FLEXURAL TEST

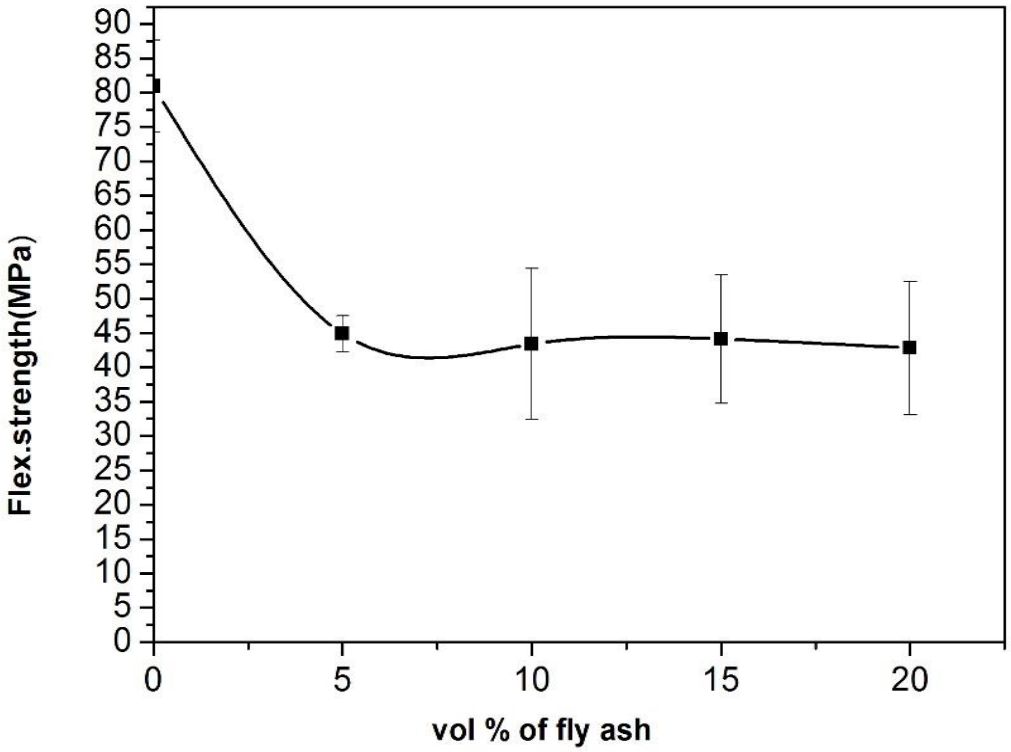
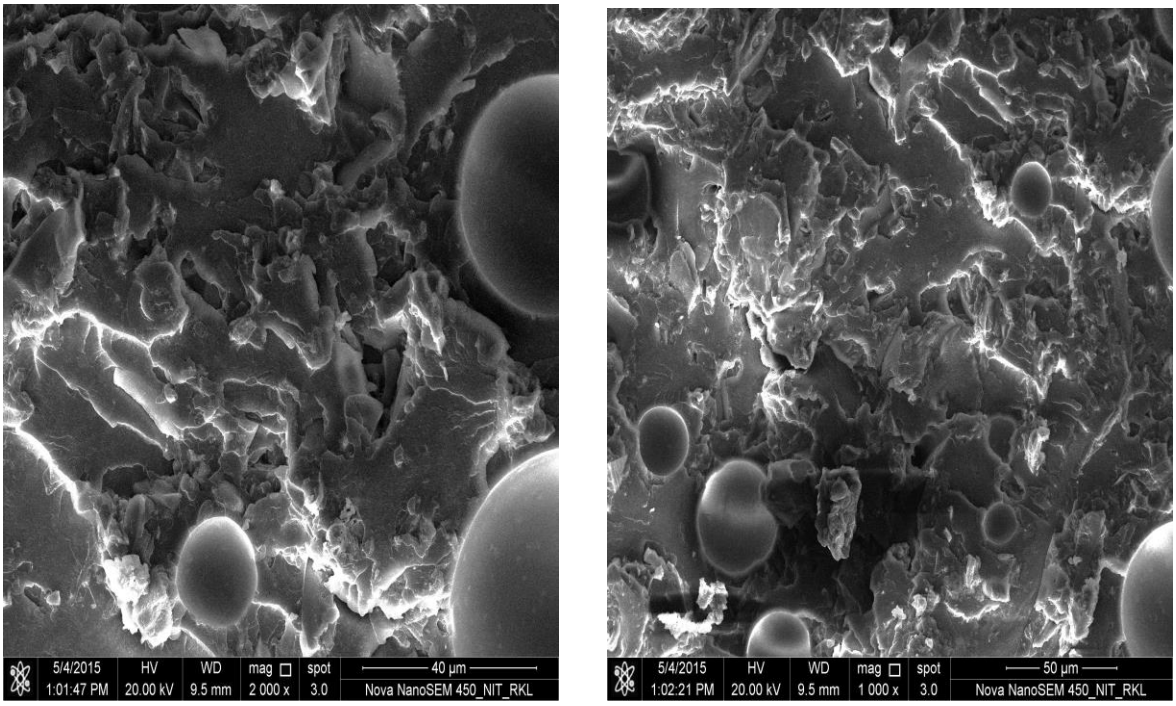


Fig- 4.11: graph between flexural Strength vs Vol% of fly ash

From the fig. 4.11 it is observed that flexural strength decreases with increase in fly ash content. It was probably caused by an incompatibility of the fly ash particles and the polymer matrix, leading to poor interfacial bonding. Fly ash particles formed cluster or agglomerate among themselves resulting in a filler-filler interaction due to the strong polarity of the hydroxyl groups on the fly ash surfaces that results in poor interfacial bonding.

4.3.4.1 MICROSTRUCTURE AFTER FLEXURAL TEST



(a) ***(b)***

Fig. 4.12 SEM images of fracture surface of 20 vol% fly ash- epoxy composite after flexural test

4.3.5 HARDNESS TEST

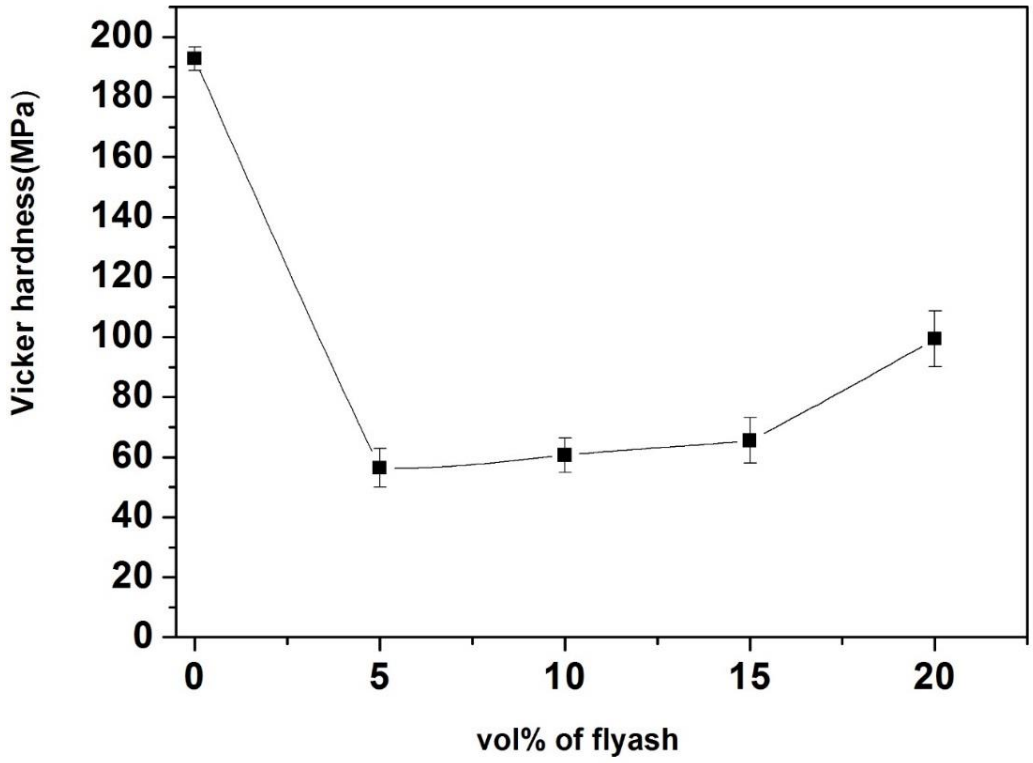


Fig- 4.13: graph between Vickers hardness vs Vol% of fly ash

From the fig- 4.13 it is observed that the hardness value deceased up to 5 vol% of fly ash, then again increased up to 20 vol% of fly ash. The reason for this type of variation of hardness along with fly ash content still needs to be explored in details.

CHAPTER 5
CONCLUSION

5.1 Conclusion

Polymer matrix composite was successfully prepared by using fly ash. The tensile strength and the compression stress are increased on addition of fly ash. The flexural strength decreases with the increase in fly ash content. The impact strength initially decreases up to 15 volume% and then it started to increase at 20 volume%. The hardness of the fly ash-epoxy composite decreased up to 5 volume%, accompanied by an increase in the hardness value with further increase in fly ash amount up to 20 volume%. Interface between fly ash and epoxy matrix plays an important role in determining the mechanical strength of the composites.

5.2 Scope for future work

This work leaves a wide scope for future research. The composites of similar nature can be tasted for different mechanical behavior. Thermoplastic also can be used apart from thermoset polymers as the matrix material and potential of industrial wastes other than fly ash can be explored.

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